



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 531

LOW IRON SAND

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, BELGIUM, THE CZECH REPUBLIC, FRANCE, GERMANY, ITALY, JAPAN, SWEDEN, TURKEY AND THE UNITED STATES OF AMERICA,
issued by the Bureau of Analysed Samples Ltd and the Society of Glass Technology.

ANALYSES

Mean of 4 values - mass content in %.

| Analyst No. | SiO ₂ | Al ₂ O ₃ | TiO ₂ | Fe ₂ O ₃ | MnO | CaO | MgO | K ₂ O | BaO | SrO | P ₂ O ₅ | Na ₂ O | Cr ₂ O ₃ | PbO | Sn | ZrO ₂ | LOI |
|----------------|------------------|--------------------------------|------------------|--------------------------------|---------|--------|---------|------------------|---------|---------|-------------------------------|-------------------|--------------------------------|---------|---------|------------------|--------|
| 1 | 99.58 | 0.0300 | 0.0173 | 0.00633 | 0.00015 | 0.0038 | 0.00120 | 0.0041 | 0.00110 | 0.00020 | 0.00070 | 0.0020 | 0.0001 | <0.0001 | ... | 0.0022 | 0.1000 |
| 2 | 99.88 | 0.0349 | 0.0148 | 0.00673 | 0.00020 | 0.0046 | 0.00133 | 0.0040 | 0.00130 | ... | ... | ... | 0.0001 | <0.001 | <0.001 | <0.01 | 0.1183 |
| 3 | 99.75 | 0.0315 | ... | 0.00668 | ... | 0.0038 | 0.00115 | ... | ... | ... | ... | 0.0037 | 0.0001 | ... | ... | ... | 0.1620 |
| 4 | 99.77 | 0.0310 | 0.0164 | 0.00593 | 0.00014 | 0.0047 | 0.00154 | 0.0032 | 0.00132 | 0.00015 | ... | 0.0035 | ... | ... | ... | 0.0027 | 0.1411 |
| 5 | 99.90 | ... | 0.0188 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 0.1038 |
| 6 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 0.0014 | 0.0618 |
| 7 | 99.78 | 0.0322 | 0.0135 | ... | 0.00020 | ... | ... | ... | 0.00108 | 0.00020 | 0.00108 | ... | 0.0002 | <0.0001 | <0.0002 | 0.0015 | 0.1113 |
| 8 | 99.79 | 0.0338 | 0.0166 | 0.00645 | 0.00014 | 0.0034 | 0.00126 | 0.0038 | 0.00113 | 0.00016 | 0.00076 | 0.0021 | 0.0001 | <0.0003 | <0.0001 | 0.0037 | 0.0673 |
| 9 | ... | ... | ... | 0.00620 | ... | ... | ... | ... | 0.00105 | ... | ... | ... | <0.0002 | <0.0001 | 0.0003 | 0.0023 | 0.1475 |
| 10 | 99.77 | 0.0346 | 0.0171 | 0.00610 | 0.00012 | 0.0042 | ... | 0.0047 | 0.00110 | 0.00028 | 0.00099 | 0.0027 | <0.0002 | <0.0005 | ... | ... | 0.1199 |
| 11 | 99.53 | 0.0355 | 0.0185 | 0.00683 | 0.00010 | 0.0035 | 0.00138 | 0.0041 | 0.00113 | 0.00020 | 0.00075 | 0.0019 | <0.0001 | <0.0001 | <0.0002 | 0.0036 | <0.10 |
| 12 | 99.60 | ... | 0.0163 | ... | ... | ... | ... | ... | ... | ... | ... | ... | <0.001 | ... | ... | ... | 0.1149 |
| 13 | 99.55 | 0.0328 | 0.0157 | 0.00663 | 0.00010 | 0.0040 | 0.0013 | 0.0040 | 0.00098 | 0.00010 | 0.00065 | 0.0026 | <0.0001 | <0.0001 | <0.0001 | 0.0011 | 0.1093 |
| 14 | 99.75 | ... | 0.0135 | 0.00605 | 0.00012 | ... | 0.00143 | 0.0039 | 0.00113 | 0.00011 | ... | 0.0035 | <0.0002 | <0.0001 | 0.0001 | 0.0030 | 0.1725 |
| 15 | 99.95 | 0.0303 | 0.0139 | 0.00623 | 0.00014 | 0.0043 | 0.00130 | 0.0035 | 0.00105 | 0.00016 | ... | 0.0050 | 0.0001 | <0.0001 | <0.0001 | 0.0029 | 0.1420 |
| 16 | ... | ... | ... | 0.00612 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 0.1000 |
| M _M | 99.74 | 0.0327 | 0.0160 | 0.00636 | 0.00014 | 0.0040 | 0.00132 | 0.0039 | 0.00112 | 0.00017 | 0.00082 | | | | | | |
| s _M | 0.14 | 0.0020 | 0.0019 | 0.00031 | 0.00004 | 0.0005 | 0.00012 | 0.0005 | 0.00011 | 0.00006 | 0.00018 | | | | | | |
| s _w | 0.04 | 0.0007 | 0.0009 | 0.00020 | 0.00002 | 0.0005 | 0.00009 | 0.0002 | 0.00006 | 0.00002 | 0.00005 | | | | | | |

Additional Information: Analyst No.8 determined Li₂O by ICP-OES and found 0.0009%.M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means. s_w: intralaboratory standard deviation.

CERTIFIED VALUES (Cv)

mass content in %

| | SiO ₂ | Al ₂ O ₃ | TiO ₂ | Fe ₂ O ₃ | MnO | CaO | MgO | K ₂ O | BaO | SrO | P ₂ O ₅ |
|--------|------------------|--------------------------------|------------------|--------------------------------|---------|--------|---------|------------------|---------|---------|-------------------------------|
| Cv | 99.74 | 0.0327 | 0.0160 | 0.00636 | 0.00014 | 0.0040 | 0.00132 | 0.0039 | 0.00112 | 0.00017 | 0.00082 |
| C(95%) | 0.09 | 0.0015 | 0.0012 | 0.00020 | 0.00003 | 0.0004 | 0.00010 | 0.0004 | 0.00007 | 0.00005 | 0.00019 |

The half width confidence interval $C(95\%) = \frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate two sided Student's t value at the 95% confidence level

for "n" acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 sections 6.1 and 10.5.2

NB: Although widely accepted within the industry "mass content in %" is neither an SI nor an IUPAC supported quantity. Multiplication of the certified value (Cv) by 10⁴ will yield the value in µg/g.

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NOTES ON METHODS USED

SILICA

All Analysts, apart from Nos. 2, 5, 7 and 12, determined silica gravimetrically; Nos. 1 and 8 after fusion with sodium carbonate and boric acid followed by coagulation with polyethylene oxide and determination of the residual silicon by ICP-OES (No. 1) and spectrophotometrically as molybdenum blue (No.8, who followed JIS M8852). Analyst No.3 followed BS 2975-2:2008 and Analyst No. 4 followed ČSN 72 0105-2 whilst Analysts Nos. 10 and 11 determined silica after digestion with hydrofluoric and sulphuric acids. Analyst No. 13 fused with sodium carbonate followed by dehydration with perchloric acid and coagulation with polyethylene oxide. No. 14 fused the sample with sodium carbonate before determining silica by double dehydration with hydrochloric acid according to ASTM C146-9a and Analyst No. 15 followed the gravimetric method in Glass Technology **14** (1973) p 5-13 in which the sample is fused with sodium carbonate and dehydrated with hydrochloric acid and the residual silica determined spectrophotometrically

Analysts Nos. 2, 5, 7 and 12 determined silica by X-Ray Fluorescence Spectrometry (XRF); No.2 according to BS EN ISO 12677, Nos. 5 and 7 after fusion with lithium tetraborate and No. 12 following the procedure in X-Ray Spectrometry **24** (1995) pages 205-218.

ALUMINA

Analyst No. 1 determined alumina by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 7, 8, 10, 11 and 15 used Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES); No.2 used microwave dissolution, No. 7 evaporated to dryness with hydrofluoric and nitric acids whilst No. 8 followed JIS M8852 and treated with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid. Analysts Nos. 11 and 15 treated the sample with hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate, No. 15 following the method in Glass Technology **34** (1993) p 239-248.

Analysts Nos. 3 and 4 used Flame Atomic Absorption Spectrometry (FAAS), No. 3 following BS 2975:1998.

Analyst No. 13 used a spectrophotometric method with Chromazurol S.

TITANIA

Analyst No. 1 determined titania by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 7, 8, 10, 11 and 14 used ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids and No. 8 after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid according to JIS M8852. Analyst No. 11 used a dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate.

Analysts Nos. 4, 13 and 15 determined titania spectrophotometrically; No. 4 with tiron according to ČSN 72 0112-2, No. 13 with chromotropic acid and No 15 according to Glass Technology **34** (1993) p 239-248.

Analysts Nos. 5 and 12 used XRF, No. 5 after fusion with lithium tetraborate and No. 12 following the procedure in X-Ray Spectrometry **24** (1995) pages 205-218.

FERRIC OXIDE

Analyst No. 1 determined ferric oxide by ICP-MS after microwave digestion.

Analysts Nos. 2, 8, 11 and 14 used ICP-OES; No. 2 after microwave dissolution, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid and No. 11 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate.

Analysts Nos. 3, 4, 9, 13 and 15 used spectrophotometric methods, No. 3 using thioglycolic acid and following BS 2975:1958 whilst No. 4 used 2,2'-bipyridyl according to ČSN 72 0110-2. Analysts Nos. 9 and 13 used ortho-phenanthroline and No.15 used the method in Glass Technology **14** (1973) p5-13.

Analysts Nos. 10 and 16 used FAAS.

MANGANESE OXIDE

Analyst No. 1 determined manganese oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

The remaining Analysts, apart from Nos. 4 and 10, used ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids and Nos. 8 and 15 after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid, No. 8 according to JIS M8852 and No 15 according to Glass Technology **34** (1993) p 239-248. Analyst No. 11 used hydrofluoric and sulphuric acids to dissolve the sample.

Analysts Nos. 4 and 10 used FAAS.

CALCIUM OXIDE

Analyst No. 1 determined calcium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 8, 10, 11 and 15 used ICP-OES; No. 2 after microwave dissolution, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid and Nos. 11 and 15 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate, No.15 following the method in Glass Technology **34** (1993) p 239-248.

Analysts Nos. 3, 4 and 13 used FAAS, No. 3 following BS 2975-2:2008.

MAGNESIUM OXIDE

Analyst No. 1 determined magnesium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 8, 11, 14 and 15 used ICP-OES; No. 2 after microwave dissolution and No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid. Analysts Nos. 11 and 15 dissolved the sample in hydrofluoric and sulphuric acids, No 15 continuing with a fusion with sodium carbonate and sodium tetraborate following the method in Glass Technology **34** (1993) p 239-248.

Analysts Nos. 3, 4 and 13 used FAAS, No. 3 following BS 2975-2:2008.

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POTASSIUM OXIDE

Analyst No. 1 determined potassium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 11, 14 and 15 used ICP-OES; No. 2 after microwave dissolution and Nos. 11 and 15 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate, No. 15 following the method in Glass Technology **34** (1993) p 239-248.

Analysts Nos. 4, 10 and 13 used FAAS.

Analyst No. 8 used flame photometry after evaporation to dryness with perchloric and hydrofluoric acids according to JIS M8852.

BARIUM OXIDE

Analyst No. 1 determined barium oxide by ICP-MS after microwave digestion.

With the exception of Analyst No 4 the remaining Analysts used ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid and Nos. 11 and 15 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate, No. 15 following the method in Glass Technology **34** (1993) p 239-248.

Analyst No 4 used FAAS.

STRONTIUM OXIDE

Analyst No. 1 determined strontium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analyst No. 4 used FAAS.

The remaining Analysts used ICP-OES; No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 acid according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric and Nos. 11 and 15 after dissolution in hydrofluoric and sulphuric acids, No 15 then by fusing with sodium carbonate and sodium tetraborate according to the method in Glass Technology **34** (1993) p 239-248.

PHOSPHORUS PENTOXIDE

Analyst No. 1 determined phosphorus pentoxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 7, 8, 11 and 13 used ICP-OES; No. 7 after evaporation to dryness with hydrofluoric and nitric acids, and No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acidic acids. Analyst No. 11 dissolved the sample in hydrofluoric and sulphuric acids.

Analyst No. 10 determined phosphorus pentoxide spectrophotometrically as phosphovanadomolybdate.

SODIUM OXIDE

Analyst No. 1 determined sodium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

*Analysts Nos. 3, 4, 10, 13 and 15 used FAAS, No 4 utilising the method in BS 2975-2:2008 and No 15 the method in Glass Technology **34** (1993) p 239-248.*

Analyst No. 8 used flame photometry after evaporation to dryness with perchloric and hydrofluoric acids according to JIS M8852.

Analysts Nos. 11 and 14 used ICP-OES, No. 11 after dissolution in hydrofluoric and sulphuric acids.

CHROMIUM OXIDE

Analyst No 1 determined chromium oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 7, 8, 10, 11, 13 and 14 used ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid and No. 11 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate.

*Analysts Nos. 3 and 15 determined chromium oxide spectrophotometrically, No. 3 with diphenyl carbazide according to BS 2975:1988 and No. 15 according to Glass Technology **34** (1993) p 239-248.*

*Analysts Nos. 9 and 12 used XRF, No. 9 using with a pressed pellet and No. 12 following the procedure in X-Ray Spectrometry **24** (1995) pages 205-218.*

LEAD OXIDE

Analyst No 1 determined lead oxide by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

*With the exception of Analyst No. 9 the remaining Analysts used ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid and No. 11 after dissolution in hydrofluoric and sulphuric acids. Analyst No. 15 used the method in Glass Technology **34** (1993) p 239-248.*

Analyst No. 9 used XRF with a pressed pellet.

TIN

With the exception of Analyst No .9 all Analyst determined tin using ICP-OES; No.2 after microwave dissolution, No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid, and No. 11 after dissolution in hydrofluoric and sulphuric acids.

Analyst No. 9 used XRF with a pressed pellet.

ZIRCONIA

Analyst No 1 determined zirconia by ICP-MS after microwave digestion in a mixture of hydrofluoric, hydrochloric and nitric acids.

Analysts Nos. 2, 6 and 9 used XRF, No. 2 according to BS EN ISO 12677 and No.9 with a pressed pellet

Analyst No. 4 determined zirconia spectrophotometrically with Arsenazo III.

*The remaining Analysts used ICP-OES; No. 7 after evaporation to dryness with hydrofluoric and nitric acids, No. 8 according to JIS M8852 and after treatment with perchloric and hydrofluoric acids, followed by nitric and hydrofluoric acids and fusion with sodium carbonate and boric acid, No. 11 after dissolution in hydrofluoric and sulphuric acids followed by fusion with sodium carbonate and sodium tetraborate and No. 15 according to the method in Glass Technology **34** (1993) p 239-248.*

LOSS ON IGNITION

All Analysts determined the loss on ignition gravimetrically by heating at 1000° ± 25° C to constant weight.

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CO-OPERATING ANALYSTS

| | | |
|-------|--|---|
| 1 | SUZUKI, Y | Ashai Glass Co., Yokohama, Japan. |
| 2 | SHAW, D., | Lucideon, Stoke-on-Trent. |
| 3 | SUNDBERG, P., | Glasforskningsinstitutet, Vaxjo, Sweden. |
| 4, 16 | BAUER, L., | Sklářský ústav Hradec Králové s. r. o. (Glass Institute Ltd.) Hradec Králové, Czech Republic. |
| 5 | FLOWER, M., | Glass Technology Services Ltd., Sheffield. |
| 6 | OLSON, S., | Guardian Glass, Carleton, USA. |
| 7 | CARPENTIER, J., | INISMa-CRIBC, Mons, Belgium. |
| 8 | TSUJINO, T., | Nippon Sheet Glass, Techno Research Co Ltd., Konoike Itami, Japan. |
| 9 | JAMIESON, S. N, <i>MSc, CChem, MRSC,</i> | NSG European Technology Centre Ltd., Lathom. |
| 10 | JONES, S.J., <i>BSc, CChem, MRSC,</i> | Ridsdale & Co. Ltd., Middlesbrough. |
| 11 | STRUBEL, C., | Schott AG, Corporate Research & Technology Development, Mainz, Germany. |
| 12 | BURTON, R.P., | Sheffield Hallam University, Sheffield |
| 13 | VERA, F., | St Gobain Recherche, Aubervilliers, France |
| 14 | SCARPA, M., | Stazione Sperimentale Del Vetro S.C.P.A. - The Glass Research Centre, Murano, Venezia, Italy. |
| 15 | YORUR, A | Research Centre, Turkiye Sis eve Cam Fabrikalari AS, Is Kuleleri Kule, Turkey. |

DESCRIPTION OF SAMPLE

BCS-CRM 531 is sold in the form of finely divided material passing a nominal 250 micron aperture and packed into bottles of 100g.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS-CRM 531 as defined in ISO Guide 34:2009 section 3.1, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006.

INTENDED USE

This sample is intended for uses such as the verification of the accuracy and repeatability of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments, for establishing values for secondary reference materials and for training purposes.

In order to ensure that a fully representative sample is taken users should take a minimum sub-sample size of 1.0g. Users of this material should be aware that the use of a smaller sub-sample size will invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 531 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. When the bottle has been opened the lid should be secured immediately after use.

TRACEABILITY

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above.

Most of the analytical methods used in the characterisation of this CRM were either international or national standard methods or methods which are technically equivalent. All laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 531 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's *t* value at the 95% confidence level for the number of results. Homogeneity has been assessed on the bulk material and has been found to be acceptable. It has not, therefore, been included in the calculated measurement uncertainty. The stability of this CRM and its transportation also make negligible contributions to the overall uncertainty of the certified values.

COMMUTABILITY

BCS-CRM 531 is intended to be used in the same physical form as that used by the participating analysts and therefore commutability is not of relevance in respect of this CRM.

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd may be obtained from the address below.

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